Data Evaluation Record on the adsorption-desorption of TSA (5-Chloro-thiazole-2-sulfonic acid sodium salt) in five soils

MRID Number 48574834

Data Requirement: OECD Guideline: 106
EPA Guideline: 835.1230

EPA PC Code: 050410
DP barcode: 403340

Test material: TSA (5-Chloro-thiazole-2-sulfonic acid sodium salt)

Primary Reviewer: Martin LeMay**,** PMRA

Secondary Reviewers: James Lin, US EPA **EPA Signature:**
Date: February 14, 2013

This study was reviewed as part of a global review. Therefore, the data evaluation was prepared in monograph form. This preface is a supplement to the attached monograph section and documents the review of the study for EFED.

Results Synopsis:
This study is classified as **supplementary,** since only one concentration of 1 mg/L was tested, instead of five as required. (Different conclusion from PMRA)

CITATION: Brands C. (2011c), Adsorption/Desorption of TSA on five soils, Makhteshim Chemical Works, Ltd. Report No.: R-28473, 20 December 2011. (MRID 48574834)

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Guideline: OECD Guideline No. 106
USEPA guideline 185.1230
Deviations: None

GLP: Fully GLP compliant (laboratory certified by Netherlands VWA Authority)

Executive Summary
The adsorption characteristics of TSA (5-chloro-thiazole-2-sulfonic acid) were studied in a batch equilibrium experiment using five soils: a silt loam [Fislis; pH 6.8, organic carbon 2.13%], a sandy loam [Sevelen; pH 7.4; organic carbon 1.61%], a loam [Hom, pH 7.2; organic carbon 2.36%], a loamy sand [Speyer 2.2, pH 5.4; organic carbon 2.16%], and a clay [Speyer 6S, pH 7.2; organic carbon 1.75%]. The adsorption coefficients were determined by equilibrating test soils with TSA at nominal concentration of 1 mg TSA/L at 20°C in the dark at soil:solution ratio of 1:1.

The recovery of the control samples ranged from 92% to 103%. Mass balances were determined for one sample of Fislis, Sevelen and Horn soil system after the adsorption cycle and ranged from 91% to 94%.

Following 48 hours of equilibration, an average of 12.2% of applied TSA was adsorbed to the Fislis silt loam, 13.0% to the Stevelen sandy loam, 13.7% to the Horn loam, 14.6% to the Speyer 2.2 loamy sand, and 7.9% to the Speyer 6S clay. The calculated K_d values were 0.15, 0.16, 0.19, 0.18 and 0.10 mL/g for Fislis, Sevelen, Horn, Speyer 2.2 and Speyer 6S, respectively. The corresponding K_{OC} were 7.1, 9.7, 8.0, 8.4 and 5.8, respectively. No significant adsorption of TSA on soil was observed; therefore, TSA is expected to be very mobile in soil.

I. MATERIALS AND METHODS

A. Materials

1. Test Materials:	5-Chloro-thiazole-2-sulfonic acid sodium salt
Description:	White powder
Lot/Batch:	231PAL080
Purity:	100%
CAS#:	
Stability of compound:	The test item was stable in the application solution during the treatment of the soil samples
2. Soil:	A summary of the physical and chemical properties of the soils is provided in **Table 1.**

Table 1: Soil Physiochemical Properties

Soil designation	soil type*	% sand	% silt	%clay	% OC	CEC (meq/ 100g)	pH
Fislis	silt loam	8.0	65.6	26.4	2.13	23	6.8
Sevelen	sandy loam	53.5	37.0	9.5	1.61	9	7.4
Horn	loam	38.7	36.2	25.1	2.36	22	7.2
Speyer 2.2	loamy sand	81.4	12.2	6.4	2.16	10	5.4
Speyer 6S	clay	21.9	36.0	42.1	1.75	22	7.2

* : USDA classification

% OC: percentage organic carbon

CEC: cationic exchange capacity

B. Study design

1. Experimental conditions

An adsorption/desorption kinetics experiment was performed to determine equilibrium time. A stock solution of 1116 mg TSA/L was prepared in Milli-Q water and was diluted in 0.01M $CaCl_2$ solution to obtain a spike solution of 10.0 mg/L. The slurries (approximately 10 g soil and 9 mL 0.01 M $CaCl_2$ solution were equilibrated at 20± 2°C in the dark for three days prior to spiking. The adsorption stage of the kinetic experiment was initiated by adding a weighted volume of approximately 1 mL of spike solution to the pre-equilibrated soil slurries. A control without soil and a blank sample of each soil were also prepared. The samples were equilibrated on a roller mixer at 20± 2°C in the dark. The slurries were centrifuged at 3, 6, 24 and 47 hours. A 1.8 mL aliquot of the supernatant was sampled for analysis. After 47 hours of equilibration, the remaining supernatant of each test system was decanted and weighted. The pH of the supernatants was measured. The remaining soil of one vessel of Fislis, Sevelen and Horn test system was stored for analysis. The desorption stage of the kinetic experiment was initiated by adding equal weight of fresh 0.01 M $CaCl_2$ to decanted soil samples and equilibrated on a roller mixer. At the desorption sampling times (3, 6, 24 and 48 hours), the slurries were centrifuged. A 1.8 mL aliquot of supernatant was sampled for analysis. After the final desorption sampling step, the remaining supernatant of the Fislis, Sevelen and Horn soils was decanted and weighted. Mass balances were determined for Fislis, Sevelen and Horn test system.

Three experiments were performed to determine the adsorption coefficient of TSA. For each test, a stock solution of TSA in 0.01M $CaCl_2$ solution or in Milli-Q water was prepared (concentration between 1060 and 1422 mg/L). The stock solutions were diluted in 0.01M $CaCl_2$ solution to obtain spike solutions of 10.0 mg/L (first and third adsorption test) or 100 mg/L (second adsorption test). The slurries (approximately 10 g soil and 9 mL 0.01 M $CaCl_2$ solution in polypropylene tubes in the first test, 100 g soil and 99 mL 0.01 M $CaCl_2$ solution in amber glass

jars in the second test, and approximately 15 g soil and 13.5 mL 0.01 M $CaCl_2$ solution in polypropylene tubes in the final test) were equilibrated overnight on a shaking device at 20 ± 2°C in the dark. After equilibration, the samples were spiked with 1 or 1.5 mL spike solution to obtain final TSA concentrations in the test solutions of 1 mg/L. A control without soil was included, as well as a blank sample of each soil (soil without test substance).

In the first test, the soil slurries were centrifuged for 5 minutes. In the second test, the slurries were poured over a paper filter (S&S 589) in a suction flask under vacuum. In the final test, the soil slurries were centrifuged for 10 minutes at 7000 rpm. After removal of the supernatant, the vials were centrifuged a second time (1 hour at 7000 rpm). The supernatants were combined. Additionally, a subsample of the supernatant was centrifuged at 15000 rpm.

2. Description of analytical procedure

The test substance concentrations in the solutions were determined by means of an LC/MS validated method. The soils were extracted with 50/50 (v/v) acetonitrile/water (20 mL) at 200 rpm for 60 minutes. The extracts were analysed by the same LC/MS method. The limit of quantification (LOQ) was assessed at 0.05 mg/l in 0.01M aqueous CaCl2.

II. RESULTS AND DISCUSSION

A. Mass Balance

The recovery for each sample was determined as the sum of the adsorption solutions and the soil extracts (non-extractables not included). The mass balance of the kinetic experiment ranged from 92% to 103%.

B. Findings

The results of the kinetic experiment showed that TSA did not adsorb significantly to the soils. The maximum adsorbed amount was 17% after 48 hours contact time with Speyer 2.2 soil. The pH of the supernatant were 7.3 (Fislis soil), 7.7 (Sevelen soil) and 7.2 (Horn soil).

The Koc values were calculated from one concentration due to the low amount adsorbed. Due to the low adsorption of TSA, no Freundlich isotherms coefficients could be determined. In the first adsorption test, K_d values ranged from 0.1 to 0.22, and K_{OC} values ranged from 5.4 to 10.4. In the second test, , K_d values ranged from 0.03 to 0.17, and K_{OC} values ranged from 1.3 to 10.4. The results of the final adsorption test was reported in **Table 2**.

Table 2: Adsorption Characteristics of TSA on Soil based on the final adsorption test

Soil designation	pH	% OC	K_d	Koc
Fislis	6.8	2.13	0.15	7.1
Sevelen	7.4	1.61	0.16	9.7
Horn	7.2	2.36	0.19	8.0
Speyer 2.2	5.4	2.16	0.18	8.4
Speyer 6S	7.2	1.75	0.10	5.8
Mean	---	---	0.16	7.8

% OC: percentage organic carbon

III. CONCLUSIONS

TSA is estimated to be very mobile in soil. The estimated K_{oc} values ranged between 5.8 and 9.7.